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Polyphenyl Ether Lubricants

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Polyphenyl ether lubricants and fluids are materials that operate within "extreme environments," and they involve applications that require lubricants, greases, or high performance fluids that can withstand extreme conditions such as high temperatures, pressures, and vacuums and are resistant to chemical and radiation attack. This chapter focuses on the current uses and some of the new developments relative to polyphenyl ethers. A few other products and/or technologies that also can be classified as falling within the preceding definition, including certain *synthetic cycloaliphatic hydrocarbons,* are discussed as well.

It has been more than 450 years since Leonardo da Vinci formulated the two basic experimental "laws" governing friction between rubbing solid surfaces. Since then, numerous publications have been developed on both lubricants and the general field of tribology, which is the "study of the phenomena and mechanisms of friction, lubrication, and wear of surfaces in relative motion." A lubricant is defined as "any material, liquid, semi-solid, solid or gas that, when interposed between two surfaces in rubbing contact, is capable of producing friction between them." Therefore, the first and foremost function of a lubricant is to reduce friction, which in turn reduces wear, saves power, reduces heat, and facilitates the formation of a load-carrying film [1].

Other important functions of lubricants, or lubricating-type materials, include:

1. Serving as a coolant, as it carries away heat from moving parts
2. Serving as an aid in sealing against foreign matter
3. Preventing corrosion and rust of steel surfaces
4. Serving as a hydraulic fluid for transmitting power
5. Acting as a vapor pump fluid, as well as a lubricant

Because lubricants may have simultaneous multiple roles, lubricant development and/or formulation to suit those roles, in a particular situation, is essential. Most importantly, lubricants must be designed to function over extended periods, without deterioration and the loss of their principal properties.

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I. HISTORICAL DEVELOPMENT

The first synthesis of polyphenyl ethers was reported in 1906 by F. Ullmann and P. Sponagel [2]. Their copper-catalyzed reaction of an alkali phenate with an aromatic halogen is the condensation reaction that is still referred to by Ullmann's name. The first commercial interest in these fluids started in the 1950s, when the U.S. Air Force funded projects aimed at developing high temperature hydraulic fluids [3]. Later, other air force contract work reported on the suitability of polyphenyl ether materials as high temperature lubricants [4].

The first large-scale application of a polyphenyl ether lubricant was in the Pratt & Whitney J-58 engines on the U.S. Air Force SR-71 aircraft. Supersonic flight generates heat from friction that cannot be removed in the conventional manner of subsonic aircraft. The designed engine lubricant temperature in this plane was a continuous 316°C, much higher than any ester-based lubricant could withstand. A five-ring polyphenyl ether base stock containing an antioxidant served as this plane's engine lubricant from 1966 until the fleet was retired from active service in 1990.

Commercial polyphenyl ether materials today include high performance fluids, lubricants, and greases. The largest application for polyphenyl ethers today is in high vacuum diffusion pumps. The study and production of vacuum was limited to the investigations of a few scientists until a little over 100 years ago [5]. After Thomas Edison had invented the lightbulb in 1879, the incandescent lamp became the first industrial product requiring a more advanced vacuum technology for its manufacture. In response to this need, improved vacuum pumps were developed. This was the beginning of modern vacuum technique. Simultaneously, vacuum processes, such as vacuum metallurgy and freeze-drying applications, were introduced. Other products followed, such as x-ray tubes, radio tubes, and, somewhat later, fluorescent lamps; more recently, semiconductive devices were developed, in particular, integrated circuits and computer chips [6].

Some of the common applications today for diffusion pumps are as follows:

1. Decorative coatings
2. Particle accelerators
3. Semiconductor manufacture
4. Electron tube manufacture
5. Sputtering
6. Mass spectrometry
7. Metallurgy
8. Leak detectors
9. Deposition systems
10. Electron microscopes
11. Space simulators
12. Optical coating

First designs of diffusion pumps by W. Gaede in Germany go back to 1915. A diffusion pump is a vapor jet pump or vapor ejector pump designed for the high vacuum regime. The only justification for calling it a diffusion pump is that the molecules of the pumped gas penetrate the vapor jet by a process similar to the diffusion of one gas into another.

A variety of organic liquids have been used as motive fluids in diffusion pumps. Criteria for selection of the fluid are as follows:

1. Low vapor pressure at room temperature
2. Good thermal stability
3. Chemical inertness

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4. Nontoxicity
5. High surface tension to minimize creep
6. High flash and fire points
7. Reasonable viscosity
8. Low heat of vaporization
9. Cost

Polyphenyl ether fluids are distinguished by exceptionally low vapor pressure, excellent thermal stability, and a tendency to wet surfaces less readily, which eliminates the "creep" that is normally associated with other materials. These fluids are used where ultrahigh vacuums of 4×10^{-10} torr, and higher, at 25°C, are required, such as in electron microscopes, mass spectrometers, and equipment for surface physics studies [7].

There are also a number of other specialty and/or niche applications for the polyphenyl ether materials. One of these exists in the formulation of radiation-resistant greases used in nuclear power plant mechanisms. Polyphenyl ethers are some of the most radiation resistant fluids available. Tests have shown γ and associated neutron radiation resistance at dosages up to 10^{10} ergs/g-C, at temperatures up to 600°F [8].

Another application of polyphenyl ethers and their derivatives is in the area of vapor phase lubricants for use in gas turbines and custom bearings [9], or where extreme environmental conditions exist. Vapor phase lubrication is achieved by heating a liquid lubricant above its boiling point. The resultant vapors are then transported to a hot bearing surface. If temperatures are below the lubricant's boiling point, the vapor condenses to provide liquid lubrication, or vapor condensation. Polyphenyl ether technology can provide improved fire safety and fatigue life, depending on the specific bearing design. In addition, the polyphenyl ethers have an advantage for this application, since they can lubricate both as a liquid at low temperatures and as a vapor at temperatures above 600°F.

II. CHEMISTRY

Polyphenyl ethers encompass a broad class of materials that have been found useful in extreme environments. In the 1950s a group of scientists at Monsanto Chemical Company (renamed Solutia Inc. in 1997) embarked on a project to develop lubricants that possessed high thermal, oxidation, and radiation stability properties, which, therefore, could be useful in extreme environments. Based on work Monsanto had done in conjunction with the U.S. Air Force, they found that polyphenyl ethers had considerable potential. They also found that unsubstituted ethers containing a high proportion of meta-linkages were the most suitable materials under these extreme conditions.

Polyphenyl ethers are long chain aromatic ethers that consist of benzene rings, or more precisely, phenyl groups, joined by oxygen atoms. The number of phenyl groups will vary, depending on the desired properties. The number of polyphenyl compounds is vast when all the substituted and unsubstituted molecules are considered, as well as the many isomer arrangements that are possible. Today, however, the commercial and semicommercial grades of the unsubstituted polyphenyl ethers are the most significant (Fig. 1). These five compounds range from two to six phenyl groups. The chemical name, abbreviated nomenclature, and trade names for each are shown in Table 1. Physical properties of these compounds are listed in Table 2.

Another important set of molecules belonging to the polyphenyl ether class of chemicals are the *polyphenyl thio/oxy ethers*. These molecules are formed by substituting sulfur for some of the connecting oxygen atoms of the ethers. Lubricants of this class were formerly designated

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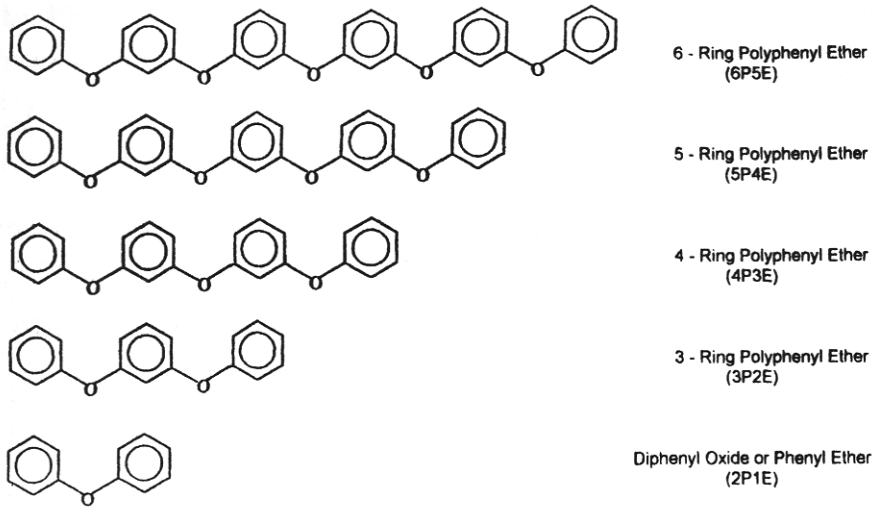

Figure 1 The polyphenyl ether family.

as C-ethers, or modified polyphenyl ethers [10,11]. Figure 2 compares the structure of a thioether and a polyphenyl ether.

Table 3 compares some properties of the polyphenyl thioethers with a five-ring polyphenyl ether (5P4E). Like many other aromatic chemicals, the oxy- and thioethers possess inherent thermal and oxidative stability, or great inertness. The oxyethers survive oxidation–corrosion tests up to 316°C. The thioethers survive tests up to 260°C and perform very well in the absence of copper up to 316°C. These aromatics also possess higher autoignition temperatures than aliphatic fluids. This property, together with their stability, allows their use at temperatures well above those of most other fluids, in applications such as high temperature jet engines, whose

Table 1 Chemical Names for Polyphenyl Ethers (PPEs)

Common and trade name[a]	Chemical name
Six-ring polyphenyl ether (6P5E) Trade name: OS-138™	Bis[*m*-(*m*-phenoxyphenoxy) phenyl] ether
Five-ring polyphenyl ether (5P4E) Trade names: Santovac® 5, 5P Ultra and OS-124™	*m*-Bis(*m*-phenoxyphenoxy)benzene
Four-ring polyphenyl ether (4P3E) Trade name: MCS-210™	Bis(*m*-phenoxyphenyl)ether
Three- and four-ring oxy- and thioethers Trade name: MCS-293™	Thiobis[phenoxybenzene] and bis(phenylmercapto)benzene
Three-ring polyphenyl ether (3P2E) Trade name: MCS-2167™	*m*-Diphenoxybenzene
Two-ring polyphenyl ether (2P1E) Trade names: Monsanto or Dow DPO	Diphenyl oxide, phenyl ether, phenoxybenzene, or diphenyl ether

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Table 2 Physical Properties of Polyphenyl Ethers (PPEs)

PPE	Form at room temperature	Pour point (°F)	Specific gravity at 24°F	Viscosity (cSt)		Autoignition temperature (°F)	Thermal stability, by isoteniscope (°F)	Flash point (°F)
				At 100°F	At 210°F			
Six-ring 6P5E	Clear liquid	65 max	1.20	2550 max	25	1170	836	600
Five-ring 5P4E	Clear liquid	45 max	1.20	360	13	1135	847	550
Four-ring 4P3E	Clear liquid	10 (−12 in mixture)	1.18	70	6	1095	825	465
Three- and four-ring oxy/thio	Hazy liquid	−20	1.20	25	4	935	693	440 min
Three-ring 3P2E	Solid	—	1.20	12	3	1070	800	380
Two-ring 2P1E	Crystal/liquid	—	1.07	2.4	1.6	1144	—	23

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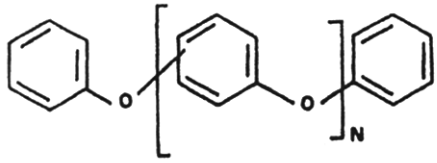

POLYPHENYL ETHERS

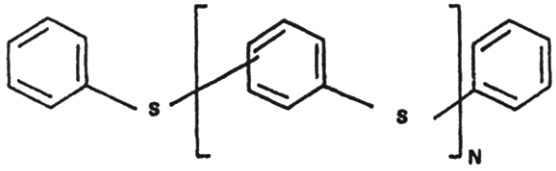

Figure 2 Structures of oxy- and thioethers.

temperature environments would prove destructive to other lubricants. As a rule, the thioether lubricants have lower pour points and viscosities than the oxyethers, depending on the number of rings and the isomer distribution.

III. PROPERTIES AND PERFORMANCE CHARACTERISTICS

Polyphenyl ethers have outstanding thermal and oxidative stability. They also have reasonable lubricity and excellent resistance to nuclear radiation and chemical attack. The process for manufacturing these materials requires many steps and involves complex raw materials. This involved process, combined with a high pour point, particularly for the higher molecular weight compounds, has limited their use to very demanding specialty applications where cost and viscosity are minor considerations, and no other product or technology can do the job [12].

A. LOW TEMPERATURE PROPERTIES

The 4P3E molecule described in Figure 1 and Tables 1 and 2 is generally formulated as a blend, to achieve a suitable material. The pure form will actually crystallize at 41°C, which is an

Table 3 Properties of Base Stocks

Property	C-Ether	PPE [5P4E]	Hindered ether
Viscosity, cSt			
At 37.8°C	25.2	363	28
At 98.9°C	4.1	13.1	5.5
At 260°C	0.81	1.2	
Thermal decomposition, °C	367	453	ca. 310
Autoignition temperature, °C	504	559	435
Pour point, °C	−29	+5	−59
Evaporative loss, % (204°C, 6.5 h)	10	<1	2.7
Surface tension, dyn/cm at 24°C	50	50	ca. 30
Isotherm secant bulk modulus, psi at 37.8°C	340,000 (0–7500 psi)	390,000 (0–6000 psi)	271,506 (0–7000 psi)

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undesirable property in a lubricant. Blending the pure molecule with other components, however, can eliminate the problem and produce a mixture with a pour point of −12°C. Below this pour point, the blended material will not crystallize, but rather becomes more glassy as the temperature continues to drop. The 5P4E molecule has similar properties, with a crystallization point of 43°C. The addition of other components will produce a mixture that will not crystallize, having a pour point of 7°C. In both cases, the pure substances will supercool and can remain in a liquid state for months at room temperature. Adding a seed crystal will hasten the eventual crystallization.

The 6P5E differs from both the 4P3E and the 5P4E in that it does not crystallize. This higher molecular weight molecule is used commercially as a pure isomer, which does not require formulating with other components. However, it generally must be heated slightly, since it has a pour point in the range of 10–15°C.

B. Thermal/Oxidation Stability

The all-meta polyphenyl ether structures have the highest thermal and oxidative stability of all other isomers of the same molecule. The differences between the isomers, however, are not large. For example, the thermal decomposition temperatures of the 4P3E isomers vary by about 8°C. Differences between the three-, four-, five-, and six-ring molecules are also small, as illustrated in Table 4. As these data show, the initial thermal stability temperatures, as measured by isoteniscope, are very high. However, the temperatures at which these fluids perform as bulk lubricants are lower. A more useful measure of thermal stability is to evaluate the increase in fluid viscosity over time while the fluid is held at a given temperature. Results are shown in Table 5 for a commercially available 5P4E product using federal test method standard 791, Method 5308, where the fluid is held for 48 hours at different temperatures using a 5 liters per hour airflow.

The inhibited 5P4E fluid shows a 100°F viscosity increase of 23% after the 48-hour test. The viscosity at operating temperature increases by a lower amount, but is not measured as a key property. The performance is considered acceptable for a bulk fluid high temperature lubricant, given that this is an accelerated test. The viscosity increase is caused by high molecular weight decomposition products. These by-products are large enough to allow distillation to economically separate them from the polyphenyl ethers, and this reclamation process is commonly employed. The decomposition products are not large enough to precipitate out of solution, however. This explains why as long as polyphenyl ethers are used at a temperature where viscosity increases are in the range of 25%, their tendency to form coking deposits is low.

C. Surface Tension

The surface tension of polyphenyl ethers is high enough that these fluids tend not to wet metal surfaces. The surface tension of the commercially available 5R4E is 49.9 dyn/cm. This property

Table 4 Thermal Decomposition Temperatures of PPEs

Polyphenyl ether	Initial decomposition temperature, by isoteniscope (°C)
4P3E	445
5P4E	465
6P5E	447

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Table 5 Thermal Stability of 5P4E

	Weight Change (mg/cm²)				
	Base fluid			Inhibited fluid	
	At 260°C	At 288°C	At 316°C	At 316°C	At 371°C
Magnesium	−0.01	+0.04	+0.25	+0.02	0.00
Aluminum	+0.01	+0.01	+0.01	+0.01	—
Titanium	0.00	+0.01	+0.02	+0.02	—
Steel	+0.01	+0.03	+0.05	+0.04	—
Copper	−0.05	+0.03	−0.95	+0.29	−0.03
Silver	+0.03	+0.06	+0.32	−0.12	−0.02
	Viscosity change (%)				
At 100°F	5	16	70	23	89
At 210°F	2	6	32	17	29

is useful when the migration of a lubricant must be avoided, such as in the lubrication of certain types of electrical components. A thin film of a polyphenyl ether is not really a thin, contiguous film, but rather a field of tiny droplets. This property tends to keep the lubricant stationary, or at least to cause it to remain in the area where lubrication is needed, rather than migrating away by spreading and forming a new surface. As a result, contamination of other components or equipment, which do not require a lubricant, is avoided. There are no other known lubricants that have this property.

D. Lubrication Characteristics

Polyphenyl ethers have good wear characteristics in bearing and gear applications. However, these materials are not recommended for piston engine applications, since no additive package has been developed, to date, to lubricate the sliding wear of piston rings. Many of the additives commonly used for this purpose in other lubricant base stocks have solubility problems in polyphenyl ethers.

Tables 6, 7, and 8 show the results of Shell four-ball wear tests, Ryder gear tests, and CRC bearing rig tests for the commercially available 5P4E fluid.

E. Vapor Pressure

The vapor pressure of the 5P4E fluid, which is commercially sold as a vacuum pump fluid, is shown in Table 9. There is nothing unique about a fluid with a low vapor pressure, which is

Table 6 Shell Four-Ball Wear Test[a]

Load (kg)	Scar diameter (mm)
10	0.80
30	0.89
50	1.13

[a] Conditions: 204°C, 600 rpm, 1 hour, 52,100 steel balls.

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Table 7 Ryder Gear Scuff Test[a]

At 75°C	2450 ± 300 ppi
At 204°C	1000 ± 150 ppi

[a] ppi, pounds per inch.

Table 8 CRC Bearing Rig Test[a]

Fluid type	Base 5P4E	Inhibited 5P4E
Bearing temperature, °C	343	343
Bulk oil temperature, °C	316	316
Oil inlet temperature, °C	288	288
Overall demerit rating	114	66
Change in 100°F viscosity	2000% (82 h)	64% (100 h)
Acid number increase	Nil	Nil
Consumption rate mL/h	39	50
Sludge formation, g	1.64	3.11

[a] CRC, Coordinating Research Council (U.S.).

Table 9 Vapor Pressure of Blended 5P4E

Pressure (mmHg)	Temperature (°C)
0.0102	260
11.8	343
194	427
760	476 (normal boiling point)

the major requirement for the working fluid in a diffusion pump. Polyphenyl ethers have found their way into this application because of a combination of a number of properties, such as superior thermal stability, exceptionally low vapor pressure, and a tendency to wet surfaces less readily and "creep" to a lesser extent than is common with other fluids. The fluid is employed for the cleanest high vacuum and ultrahigh vacuum applications, where its excellent high vacuum performance and low tendency to migrate into the pump system are crucial. Since the fluid is chemically stable, noncorrosive, safe, nontoxic, and has excellent lubricating properties, it is so used to lubricate mechanisms in the overall vacuum system [13]. Since diffusion pumps work by boiling their fluid with an electric heating element, there are conditions under which the fluid will experience high surface temperatures.

Another useful property of polyphenyl ethers in this application is their ability to resist tack from other chemicals that may be introduced into the vacuum chamber. Acids, bases, and halogens have little effect on polyphenyl ethers.

IV. MANUFACTURE, MARKETING, AND ECONOMICS

There continues to be only one producer of polyphenyl ethers in the United States. Monsanto Company began to manufacture and market this family of products worldwide in the 1950s.

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The entire business was sold to Santovac Fluids, Inc., in early 1997. Santovac Fluids, however, was very familiar with this technology because it had been assisting Monsanto over a period of about 20 years in developing and optimizing the manufacturing process for polyphenyl ethers and related materials, through one of its affiliated companies, Findett Corporation. The largest volume product continues to be the 5P4E, which is used as a jet lubricant and as a vacuum pump fluid. Smaller quantities of the other polyphenyl ethers have been produced either under contract or for proprietary applications.

A few other companies have attempted to manufacture polyphenyl ethers in other countries. However, they either have abandoned their efforts or, apparently, are producing the compounds for captive use.

The market price for polyphenyl ethers is relatively high compared to other synthetics. This is due to expensive raw materials, a complex, multistep manufacturing process, and small markets, which are characterized as having extreme environments that require products with unique properties. In general, the price of the polyphenyl ethers is only one factor that enters into the decision to use these products. Polyphenyl ethers are chosen either because they have properties that no other material possesses, or because the benefits far outweigh the costs compared to alternative technologies in a specific application.

V. OUTLOOK

The outlook for the future of polyphenyl ethers is promising, since there is a growing demand for lubricants, greases, and fluids that can function in extreme environments. The trend toward smaller and lighter automobiles will continue, and as a result, under-the-hood temperatures will rise beyond the useful operating range of standard hydrocarbon-type lubricants and greases. Therefore, polyphenyl ethers and related materials that possess high temperature properties will be a viable option. The same scenario exists in both the aircraft and industrial markets, where higher temperatures and pressure/vacuum environments will be influencing new and improved designs.

Another class of materials falling within the definition of ‘ ‘extreme environment” materials are *synthetic cycloaliphatic hydrocarbons,* which are used as traction lubricants, fluids, and greases. These materials become solid under high stress and high sheer, and are sometimes referred to as “superfluids.” They are liquid beyond the contact area. The chemical structures of these cycloaliphatic hydrocarbon materials have been described in a number of patents [14,15]. The materials differ from other molecules in that they combine the unique properties of elastohydrodynamic (EHD) lubrication with a very high coefficient of traction C_t. This association appears to be optimized in these types of hydrocarbon material and is explained to some degree by the molecular structure and the manner in which the molecule functions, or how the molecule becomes “packed” when it is placed under high pressure and/or high sheer/stress. This makes EHD fluids very useful in traction drives.

Development of new traction drives for automotive, industrial, and aircraft applications created renewed interest in traction lubricants. These drives transmit power efficiently at lower manufactured cost. They include continuously variable transmission (CVT) drives under development by automotive companies in the United States, Europe, and Japan. Prototypes of CVT drives are currently being tested, and more advanced fleet testing will take place over the next few years [16].

During the 1960s, Monsanto started doing research into pressure-stiffening lubricants. This work, which continued for a number of years, resulted in the development of unique fluids that possessed a relatively high C_t. Unlike most commercial fluids known, these synthetic hydrocarbon materials are lubricants at normal pressures and shear rates, but turn instantly to a glassy

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pseudosolid as the pressure at the point of contact increases. Release of the pressure restores the fluid to its liquid state. This unique property makes these synthetic hydrocarbons very useful for traction drive applications and other end uses where extreme pressure and fatigue life are issues.

Early theories assumed that metal-to-metal contact was the mechanism that transmitted force in rolling contact, with the lubricant acting primarily as a coolant. However, experimental measurements of the interfacial film thickness show that a distinct lubrication regime, the elastohydrodynamic, exists between hydrodynamic and boundary lubrication. It is the combination of physical–chemical properties of the lubricant in this regime that controls the performance of traction drives.

Several theories were advanced to explain the EHD property of a traction lubricant. The leading one suggests that under high contact stress (pressure) and high shear rates of rolling contact, the viscosity of the lubricant film in the contact area increases to a glassy solid state. Thus, this "pad" of solid film transmits the tangential force with a shear resistance far beyond the capability of a liquid film. The transitory, solid "pad" protects parts by preventing metal-to-metal contact; and when compressed in surface cracks, it inhibits fatigue crack propagation.

Freed from the rolling contact area, such a lubricant returns immediately to its liquid state and normal physical properties. The coefficient of traction has been shown to be independent of normal viscosity, viscosity index, pressure viscosity, specific gravity, or other common physical properties. Thus, the more "solid" the film's momentary "pad" can become under the pressure exerted, the higher will be its coefficient of traction and the more power it will transmit between rolling elements for a given normal contact load. Figure 3 depicts the relationship between power (torque) and the forces associated with rolling contact surfaces.

The output of a traction drive of a given design is limited primarily by the coefficient of traction of the lubricant. Traction lubricants with the highest coefficient of traction make it possible to develop the highest output capability with any type of traction drive. Typically, variations in normal load, rolling speed, and temperature all have some effect on a lubricant's traction coefficient, depending on the design of the drive and the spinning motion in the contact area.

It has been shown that molecular geometry has an important effect on the coefficient of traction. The ability of molecules to arrange themselves into an apparent glassy state under high shear increases the coefficient of traction.

Table 10 shows coefficient of traction data for a number of petroleum and synthetic-based fluids. Calculations based on these results show cycloaliphatic hydrocarbons to have coefficients of traction that are at least 60% higher than those previously reported for naphthenic oils or other fluids.

A high coefficient of traction is an inherent property of a lubricant. It is related to the shape and behavior of the lubricant molecules. Lubricants are chemically designed to have shaped molecular structures to produce high coefficients of traction.

Commercial traction lubricants have been developed based on cycloaliphatic hydrocarbons, and these have been used successfully in industrial drives and automotive and aircraft continuously variable transmission drives. The CVT traction lubricants meet a broader range of lubrication requirements over a wide temperature range (−40 to +150°C) and provide enhanced thermal and oxidative stability and materials compatibility.

Although commercial synthetic traction lubricants are used worldwide today, more work is required to develop products to meet all the requirements for industrial equipment and automotive transmission designs in the twenty-first century. The main driving forces will be reduced equipment maintenance, stricter government regulations, and improved performance and energy efficiencies.

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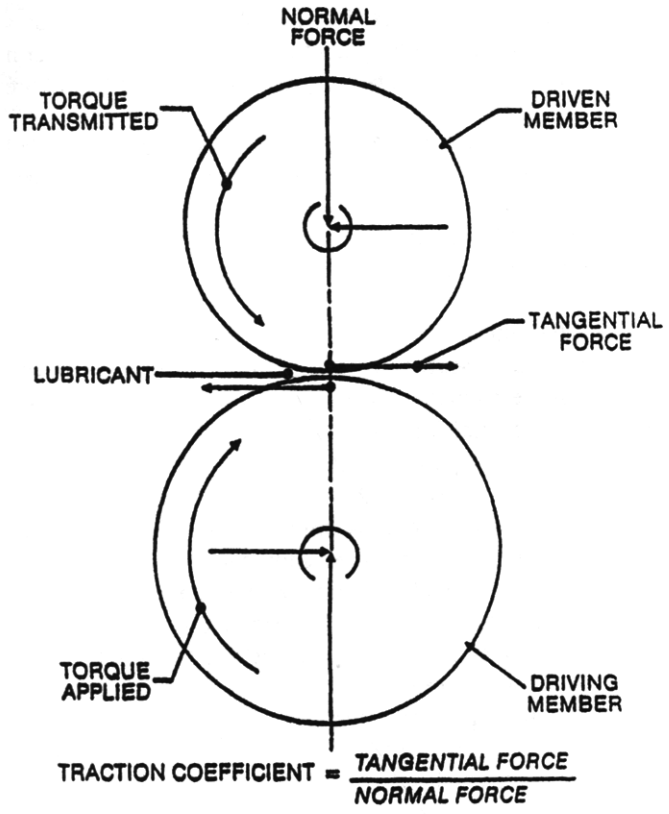

Figure 3 Transfer of power (torque) through a lubricant film.

Table 10 Coefficient of Traction Data for Various Fluids[a]

Lubricant type	Coefficient of traction, C_t
Cycloaliphatic hydrocarbon	0.105
Naphthenic oil	0.065
Polyphenyl ether (5P4E)	0.064
Perfluoropolyether	0.060
Naphthenic mineral oil	0.045
Synthetic ester oil	0.035
Poly(α-olefin) (PAO)	0.020

[a] The coefficient of traction C_t was measured on a twin disk machine at 40°C, 300,000 psi peak Hertz pressure.

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